

DTIC FILE COPY

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SECURITY CLASS

DOCUMENTATION PAGE

1a. REPORT:

AD-A198 227

2a. SECURITY:

2b. DECLASSIFICATION/DOWNGRADING SCHEDULE

4. PERFORMING ORGANIZATION REPORT NUMBER(S)

Technical Report #11
Texas A&M University

6a. NAME OF PERFORMING ORGANIZATION

Texas A & M University

6b. OFFICE SYMBOL
(If applicable)

6c. ADDRESS (City, State and ZIP Code)

College Station, TX 77853

8a. NAME OF FUNDING/SPONSORING
ORGANIZATION

AFOSR

8b. OFFICE SYMBOL
(If applicable)

NM

8c. ADDRESS (City, State and ZIP Code)

Bldg 410
Bolling Air Force Base
Washington, DC 20332

1b. RESTRICTIVE MARKINGS

3. DISTRIBUTION/AVAILABILITY OF REPORT

Approved For Public Release,
Distribution Unlimited

5. MONITORING ORGANIZATION REPORT NUMBER(S)

AFOSR-TR- 88-0843

7a. NAME OF MONITORING ORGANIZATION

Air Force Office of Scientific Research

7b. ADDRESS (City, State and ZIP Code)

Same as SC

9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER

AFOSR-649620-85-C-0144

10. SOURCE OF FUNDING NOS.

PROGRAM
ELEMENT NO.PROJECT
NO.TASK
NO.UNIT
NO.611021
A QUICK AND EASY MULTIPLE USE CALIBRATION CURVE
PROCEDURE

2304

AB

MAR 29 1988
90 E

12. PERSONAL AUTHOR(S)

Carroll, R.J., Sacks, J. and Spiegelman, C.H.

13a. TYPE OF REPORT

Technical Journal

13b. TIME COVERED

FROM 8/87 TO 8/88

14. DATE OF REPORT (Yr., Mo., Day)

15. PAGE COUNT

18

16. SUPPLEMENTARY NOTATION

17. COSATI CODES

FIELD GROUP SUB. GR.

18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)

Scheffe uncertainty bounds, confidence intervals,
nonparametric calibration, calibration curves. (myu)

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

The standard multiple use calibration procedure due to Scheffe (1973) states that with probability 1- α , the proportion of calculated confidence intervals containing the true unknowns is at least 1- α in the long run. The probability 1- α refers to the probability that the calibration experiment results in a 'good' outcome. In Scheffe's formulation a good outcome involves both coverage of the true underlying regression curve and an upper confidence limit for θ , the scale parameter. Scheffe's procedure is fairly difficult for practitioners to apply because it relies on tables that are not easy to use. A simpler notion of 'goodness' which only requires the calibration experiment to result in coverage of the underlying regression leads to easily calculated confidence intervals for the unknowns. In addition, these intervals are generally shorter than Scheffe's. An application example is given to illustrate the technique.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT

UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT. ☐ DTIC USERS ☐

21. ABSTRACT SECURITY CLASSIFICATION

UNCLASSIFIED

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22c. OFFICE SYMBOL

NM

AFOSR-TR. 88-0843

**A QUICK AND EASY MULTIPLE USE
CALIBRATION CURVE PROCEDURE**

Technical Report # 11

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A QUICK AND EASY MULTIPLE USE CALIBRATION CURVE PROCEDURE

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Key words: Scheffe uncertainty bounds, confidence intervals, nonparametric calibration.

ABSTRACT

The standard multiple use calibration procedure due to Scheffe (1973) states that with probability $1-\delta$, the proportion of calculated confidence intervals containing the true unknowns is at least $1-\alpha$ in the long run. The probability $1-\delta$ refers to the probability that the calibration experiment results in a 'good' outcome. In Scheffe's formulation a good outcome involves both coverage of the true underlying regression curve and an upper confidence limit for σ , the scale parameter. Scheffe's procedure is fairly difficult for practitioners to apply because it relies on tables that are not easy to use. A simpler notion of 'goodness' which only requires the calibration experiment to result in coverage of the underlying regression leads to easily calculated confidence intervals for the unknowns. In addition, these intervals are generally shorter than Scheffe's. An application example is given to illustrate the technique.

1. Introduction

A calibration curve is often used to relate instrument readings to established standards and thereby provide meaningful measurements. Accounting for the resulting uncertainty from calibration is of central importance. A feature of critical interest is how often the calibration curve is to be used. Most often, in our experience, the calibration curve will be used many times. Consequently, the measurements will be correlated and this dependence must be accounted for. Calibration procedures that account for such dependence have been developed for the linear model by Lieberman, Miller, and Hamilton (1967)

and Scheffe (1973). In this article we present a new method for coping with multiple use calibration. Our method is based on a modification of the Scheffe (1973) confidence statements, but the result is far simpler to implement and generally leads to shorter intervals. Intervals akin to ours have been used by scientists and engineers for some time without theoretical justification for example, see Hockersmith and Ku (1964).

Consider for example the simple linear regression model. Assume that the responses y are linearly related to predictors x with slope β_1 and intercept β_0 , and that the responses are normally distributed with standard deviation σ . Symbolically, we write the model as

$$(1.1) \quad y = \beta_0 + \beta_1 x + \epsilon$$

$$\epsilon \sim N(0,1).$$

A training sample (or calibration experiment) of size N is used to construct the classical least squares estimates $(\hat{\beta}_0, \hat{\beta}_1, \hat{\sigma})$ of $(\beta_0, \beta_1, \sigma)$ and then the calibration curve $\hat{\beta}_0 + \hat{\beta}_1 x$ is formed with $\hat{\sigma}S(x)$ denoting the estimated standard deviation of the curve. The standard deviation of $\hat{\beta}_0 + \hat{\beta}_1 x$ is defined to be $\sigma S(x)$, the estimated standard deviation is obtained if σ is replaced by $\hat{\sigma}$. We denote the observations (y_1, \dots, y_N) of the experiment by \underline{Y} and use \underline{y} to denote a specific outcome of \underline{Y} .

After this calibration experiment is performed and the calibration curve is constructed, we typically observe a series of new "responses" y_1^*, y_2^*, \dots which satisfy (1.1). The associated true values x_1^*, x_2^*, \dots are not observed but are to be estimated and confidence intervals for x_1^*, x_2^*, \dots are to be constructed.

The usual estimate is $\hat{x}_j = (\hat{y}_j^* - \hat{\beta}_0)/\hat{\beta}_1$, and the remaining problem concerns confidence intervals. In what follows, it is convenient to write the true curve as $f(x) = \beta_0 + \beta_1 x$ and the constructed calibration curve as $\hat{f}(x) = \hat{\beta}_0 + \hat{\beta}_1 x$. The calibration region or the set of x 's of interest will be denoted by I .

There is fairly broad agreement on what to do in the case that a single unknown x^* is to be estimated based on y^* . A confidence interval for x^* can be obtained as follows: For given x , let $J(x)$ be a $(1-\alpha) \cdot 100\%$ prediction interval for the response y , i.e.,

$$J(x) = \hat{f}(x) \pm t_{N-2}(1-\alpha/2) \hat{\sigma}(1+S^2(x))^{1/2},$$

where $t_{N-2}(1-\alpha/2)$ is the $1-\alpha/2$ percentile of the Student t -distribution with $N-2$ degrees of freedom. For any given y , let $K(y)$ be the set of x for which y is "correctly" predicted in the sense of being in the prediction interval, i.e., $K(y) = \{x | y \in J(x)\}$. Then we know that $K(y^*)$ is a valid $1-\alpha$ confidence interval for x^* . In symbols,

$$\Pr\{x^* \in K(y^*)\} = \Pr\{y^* \in J(x^*)\} = 1-\alpha.$$

Difficulties arise when one wishes to estimate a sequence of unknowns x_1^*, x_2^*, \dots from observations y_1^*, y_2^*, \dots . For this multiple use case, Scheffe (1973) suggests modifying the prediction bands to be of the form

$$(1.2) \quad J_S(x) = \{y | \hat{f}(x) - \hat{\sigma}(c_1 + c_2 S(x)) \leq y \leq \hat{f}(x) + \hat{\sigma}(c_1 + c_2 S(x))\}.$$

These generally lead to confidence intervals for x_j^* given by $K_S(y_j^*) = \{x | y_j^* \in J_S(x)\}$; rarely in practice are these sets disconnected. The constants c_1 and c_2 have to be chosen to satisfy uncertainty criteria required of the confidence intervals.

The Scheffe method, as does ours, involves an uncertainty statement that encompasses two sources of error: one from the calibration experiment, the other from the post calibration measurements. The data from the calibration experiments are used to estimate f and σ and one measure of uncertainty is the probability of the calibration experiment producing a 'good' estimate of f and σ . Formally, Scheffe defines a 'good' set by

$$(1.3) \quad G_S = \{y | |\hat{f}(x) - f(x)| \leq c_2 \hat{\sigma} S(x) \text{ all } x \in I \text{ and } \hat{\sigma}/\sigma \geq b\}$$

where b and c_2 are chosen so that the probability that the experiment is good is $1-\delta$ that is,

$$(1.4) \quad P_{\beta, \sigma^2}[\underline{Y} \in G_S] \geq 1-\delta, \text{ all } \beta, \sigma^2$$

(here $\beta = (\beta_0, \beta_1)$). The connection between b , c_2 and the c_1 , c_2 of (1.2) and α , δ is spelled out in Scheffe (1973) and we do not repeat the details. The meaning of (1.3) and (1.4) is clear: 'good' refers to f covered and σ bounded and the probability of 'good' outcomes is controlled to be at least $1-\delta$.

The other level of uncertainty involves the probability of covering the true x^* when a new observation y^* is made. Using the intervals defined at (1.2) Scheffe requires that, given a 'good' outcome, the probability of covering

the true x^* is at least $1-\alpha$. In formal terms

$$(1.5) \quad P_{\beta, \sigma^2, x^*} [K_S(y^*) \supset x^* \mid \underline{Y} = \underline{y}] \geq 1-\alpha$$

for all β, σ^2 , all $x^* \in I$, all $\underline{y} \in G_S$.

If (1.5) holds then the long-run proportion of confidence intervals covering the true unknowns $\{x_j^*\}$ will be at least $1-\alpha$ if the calibration experiment resulted in an outcome in G_S . The choice of b and the inclusion of a bound on σ in the definition of G_S is to allow the calculation leading to (1.5).

The choice of c_1 and c_2 that assures both (1.4) and (1.5) can be obtained from tables in Scheffe's paper. However, as noted in Lechner, Reeve and Spiegelman (1982), this table is not easy for practitioners to use. The difficulty is the need to compute the minimum and maximum of $S(x)$ over the region I of interest and this can be a formidable task in linear models more general than (1.1).

Our approach is first define the 'good' set not by G_S but, by

$$(1.6) \quad G = \{ \underline{y} \mid |\hat{f}(x) - f(x)| \leq c_2 \hat{\sigma} S(x), \text{ all } x \in I \},$$

where c_2 is to be chosen to satisfy (1.8) below. This restricts attention to whether the true f is covered; $\hat{\sigma}$ is auxiliary to that concern. In Section 2 we show how this modification leads to a substantial simplification in the calculation of confidence intervals for the unknown $\{x_j^*\}$. In fact, we take the c_1 and c_2 of (1.2) as

$$(1.7) \quad \begin{aligned} c_1 &= t_{N-2}(1-\alpha/2) \\ c_2 &= (2F_{2,N-2}(1-\delta))^{1/2} \end{aligned}$$

($F_{2,N-2}(1-\delta)$ is the $1-\delta$ quantile of the indicated F distribution).

To get the second part of the uncertainty statement note that the choice of c_2 in (1.7) results in

$$(1.8) \quad P_{\beta, \sigma^2}[Y \in G] \geq 1-\delta \quad \text{for all } \beta, \sigma^2.$$

If we let

$$Z = \max_{x \in I} |\hat{f}(x) - f(x)|/S(x),$$

so that G can be restated as $\{Z \leq c_2 \hat{\sigma}\}$, then the intervals defined through (1.2) with c_1, c_2 taken from (1.7) satisfy

$$(1.9) \quad P_{\beta, \sigma^2, x^*}[K(y^*) \supset x^* | Z = z, G] \geq 1 - \alpha$$

for all $\beta, \sigma^2, x^* \in I$, and z . Note that the choice c_1, c_2 of (1.7), or any choice permitting (1.8) and (1.9) to hold, will differ from the c_1, c_2 that satisfy (1.4) and (1.5).

In our experience in practical case we get narrower intervals by our method than by Scheffe's. This is borne out in the example in Section 3 below. Which is more appropriate, G or G_S , (1.5) or (1.9), for uncertainty statements is not clear on prior grounds. Both formalize the long run conduct of post-calibration experiments in terms of the outcomes of the initial calibration

experiment. The simplicity of (1.7) and its consequences (1.8) and (1.9) appear to us to have a decided advantage - practitioners understand the role of the F and the t distributions and have immediate access to appropriate tables, the uncertainty statements of (1.8) and (1.9) have little added conceptual complications, and there are no extra computational difficulties in obtaining the needed c_1, c_2 . Indeed, there is a simple graphical explanation in Figure 1 which shows how the confidence band on f and the error in y^* combine to produce the desired interval for x^* . In Figure 1 the $(1-\delta)$ confidence band is given by the outer curves; on the y -axis there is a $(1-\alpha)$ interval with y^* in the center. By reflecting the endpoints of the latter interval through the confidence band we get two points that surround \hat{x}^* and define the confidence interval for x^* . For these reasons of simplicity and utility we are compelled to recommend use of G and the consequent easy and quick use of (1.7).

The choices of c_1 and c_2 can be improved at the expense of added analysis and computation (see Remarks 1 and 2 of Section 2). If the model (1.1) is replaced by a multiple regression with p variables including the intercept, then $c_1 = t_{N-p}(1-\alpha/2)$ and $c_2 = (pF_{p,N-p}(1-\delta))^{1/2}$.

2. Derivation of Results

Assume f follows a multiple regression model with p parameters. As noted in (1.6) of Section 1 we regard a good outcome of the calibration experiment as one that is in G . Our first step is to call on standard least squares theory and note that the requirement

$$P[G] = P[|(\hat{f}(x) - f(x))/S(x)| \leq c_2 \hat{\sigma} \text{ all } x \in I] \geq 1-\delta$$

is fulfilled if $c_2 = (p F_{p, N-p}(1-\delta))^{1/2}$ (this can be seen in Scheffe, 1973). Ideally we would like the smallest possible c_2 but the present choice is simple and adequate for now. We note that Scheffe uses a multiple γ of this c_2 for some $.95 \leq \gamma \leq 1.2$ whose choice depends on $S(x)$, p , N .

Recall from Section 1 that G can be expressed as $\{y|Z \leq c_2 \hat{\sigma}\}$ where Z is defined following (1.8). The independence of \hat{f} and $\hat{\sigma}$, which comes from the assumption of normality, implies that Z and $\hat{\sigma}$ are independent random variables. This independence and straightforward calculation produces

$$\begin{aligned} (2.2) \quad & P[Z \leq c_2 \hat{\sigma}, \hat{\sigma} \geq q \mid Z = z] \\ &= P[z \leq c_2 \hat{\sigma}, \hat{\sigma} \geq q] \\ &= \min [P[\hat{\sigma} \geq z/c_2], P[\hat{\sigma} \geq q]] \\ &\geq P[\hat{\sigma} \geq z/c_2] P[\hat{\sigma} \geq q] \\ &= P[Z \leq c_2 \hat{\sigma} \mid Z = z] P[\hat{\sigma} \geq q]. \end{aligned}$$

By dividing the two ends of (2.2) we get

$$(2.3) \quad P[\hat{\sigma} \geq q \mid Z \leq c_2 \hat{\sigma}, Z = z] \geq P[\hat{\sigma} \geq q].$$

Let $A_z = \{Z = z, Z \leq c_2 \hat{\sigma}\}$. Then

$$P[K(y) \supset x \mid A_z] = P[\hat{f}(x) - f(x) - c_2 \hat{\sigma} S(x) - \hat{\sigma} c_1 \leq \sigma \leq \hat{\sigma} c_1 + \hat{f}(x) - f(x) + c_2 \hat{\sigma} S(x) \mid A_z]$$

$$(2.4) \quad \geq P[-\hat{\sigma} c_1 \leq \sigma \leq \hat{\sigma} c_1 \mid A_z] = \int (\phi(c_1 t) - \phi(-c_1 t)) dH(t)$$

where H = the conditional distribution of $\hat{\sigma}/\sigma$ given A_z . The integrand in (2.4) is clearly increasing in t and we have just shown in (2.3) that

$$H(t) \leq P[\hat{\sigma}/\sigma \leq t].$$

Hence the right side of (2.4) is at least as large as

$$\int (\phi(c_1 t) - \phi(-c_1 t)) d P[\hat{\sigma}/\sigma \leq t] = P[|t_{N-p}| \leq c_1].$$

Choose $c_1 = t_{N-p}(1-\alpha/2)$ and obtain the result: given Z, G the probability of covering the unknown x is at least $1-\alpha$.

Remark 1. The use of $c_2 = (pF_{p, N-p}(\delta))^{1/2}$ is only made for simplicity. It is known (Wynn and Bloomfield, 1971; Knafl, Sacks, and Ylvisaker, 1985) that better choices of c_2 can be made to provide statements like (1.8). An immediate consequence of the arguments here is that such a c_2 combined with $c_1 = t_{N-p}(1-\alpha/2)$ also provides useable confidence bounds and narrower ones than described above.

Remark 2. Examination of (2.4), especially going from the second to the third expression, reveals that improvement can be obtained by using the monotonicity properties of the t distribution to replace the third term by

$$P[-2\hat{\sigma}S(x)c_2 - c_1\hat{\sigma} < c_1\hat{\sigma} | y].$$

This leads to choosing c_1 to satisfy

$$(2.5) \quad P[-c_1 - 2c_2 S(x) < t_{N-p} < c_1] = 1 - \alpha.$$

Finding c_1 to satisfy (2.5) requires knowledge of $S(x)$. Since the x 's are unknown, this is unrealistic but we can replace $S(x)$ by $\min_x S(x) = S_0$ (say) and then use t-tables to find c_1 so that

$$P[-c_1 - 2c_2 S_0 < t_{N-p} < c_1] = 1 - \alpha.$$

Unless $S_0 = 0$, such a c_1 is smaller than $t_{N-p}(1-\alpha/2)$. A less precise but practical approach is to replace $S(x)$ by $S(\hat{x})$ where $\hat{x} = \hat{f}^{-1}(y)$ and treat $S(\hat{x})$ in (2.5) as if it were non-random. This gives data-dependent c_1 but leads to nominal values of confidence that should be close to correct.

Remark 2 is a relatively simple computation and its implementation can lead to substantial savings in the lengths of intervals. This is clearly indicated in the example presented in Section 3, see Table 2. We do not over-emphasize Remarks 1 and 2 because our concern is to make clear the advantage of the quick and easy use of (1.7). Our recommendation overall would be to employ Remark 2 as well because it is relatively easy to carry out. Implementing Remark 1 is unlikely to yield great benefits unless the degree p is bigger than 1 or the problem is one where nonparametric calibration (see Remark 4 below) is needed. In such cases it would be necessary to incorporate substantial computations (see Knafl, Sacks, Spiegelman, and Ylvisaker, 1984; Knafl, Sacks, and Ylvisaker, 1985) and would belie our use of the words "quick and easy."

Remark 3. It is straightforward to adjust the statements and procedure if the calibration experiment is performed in one laboratory but the calibrations

are carried on elsewhere. This situation leads possibly to two σ 's, one in the calibration experiment, the other from new observations y^* . Under usual assumptions of independence, the modifications needed are easy to make.

Remark 4. (Nonparametric calibration.) Knafl, et al. (1984) discuss calibration when the mean of the responses $f(x)$ does not follow a strict linear model so that the use of a multiple regression as done above would be inappropriate. Examination of the details of arguments used in that paper shows that considerable simplification in computation can result by use of the G of (1.6). We forego elaboration of the necessary technicalities.

3. Example

A simple example arises in atomic absorption spectroscopy for which there are twelve data points as shown in Table 1. Figure 2a is a plot of the data of Table 1. Figure 2b is a plot of the residuals from a straight line fit. These figures indicate that a straight-line fit to the data is entirely reasonable. We chose $\alpha = 0.10$ and $\hat{\sigma} = 0.003$. Following Scheffe we would get calibrations based on $\hat{\sigma} = 0.003$, $c_1 = 2.32$ and $c_2 = 2.36$, see the discussion after equation (1.6). As an exercise, the reader may wish to attempt the calculation of the Scheffe constants and compare the effort involved with that required to look up our $c_1 = t_{10}(0.95) = 1.81$ and $c_2 = (2F_{2,10}(0.90))^{1/2} = 2.41$. The calibration intervals are given in Table 2 for selected values of y ; these intervals provide numerical evidence of our claim that the Scheffe intervals are typically significantly longer. The difference in lengths is due to the difference between G and G_S in (1.6) and (1.3).

4. Acknowledgement

The authors thank Joan Rosenblatt and Keith Eberhardt for their helpful comments and Robert Watters for providing the data used in the example of Section 3.

The research by Carroll was supported by AFOSR Contract AFOSR-F-49620-85-C-0114. The research by Sacks was supported in part by ONR Contract N00014-85-K-0357 and NSF Grant DMS-85-03793. Spiegelman's research was supported by ONR Contract N00014-83-K-0005 and NR-042544.

Table 1
Atomic Absorption Spectroscopy

x	y
0.0	.045
0.0	.047
0.0	.051
0.0	.054
.050	.084
.050	.087
.100	.115
.100	.116
.200	.183
.200	.191
.500	.395
.500	.399

x = amount of copper in a dilute
acid solution measured in
micrograms/milliliter.

y = instrument response in
absorbance units.

(The data have been rounded to three decimal places.)

Table 2

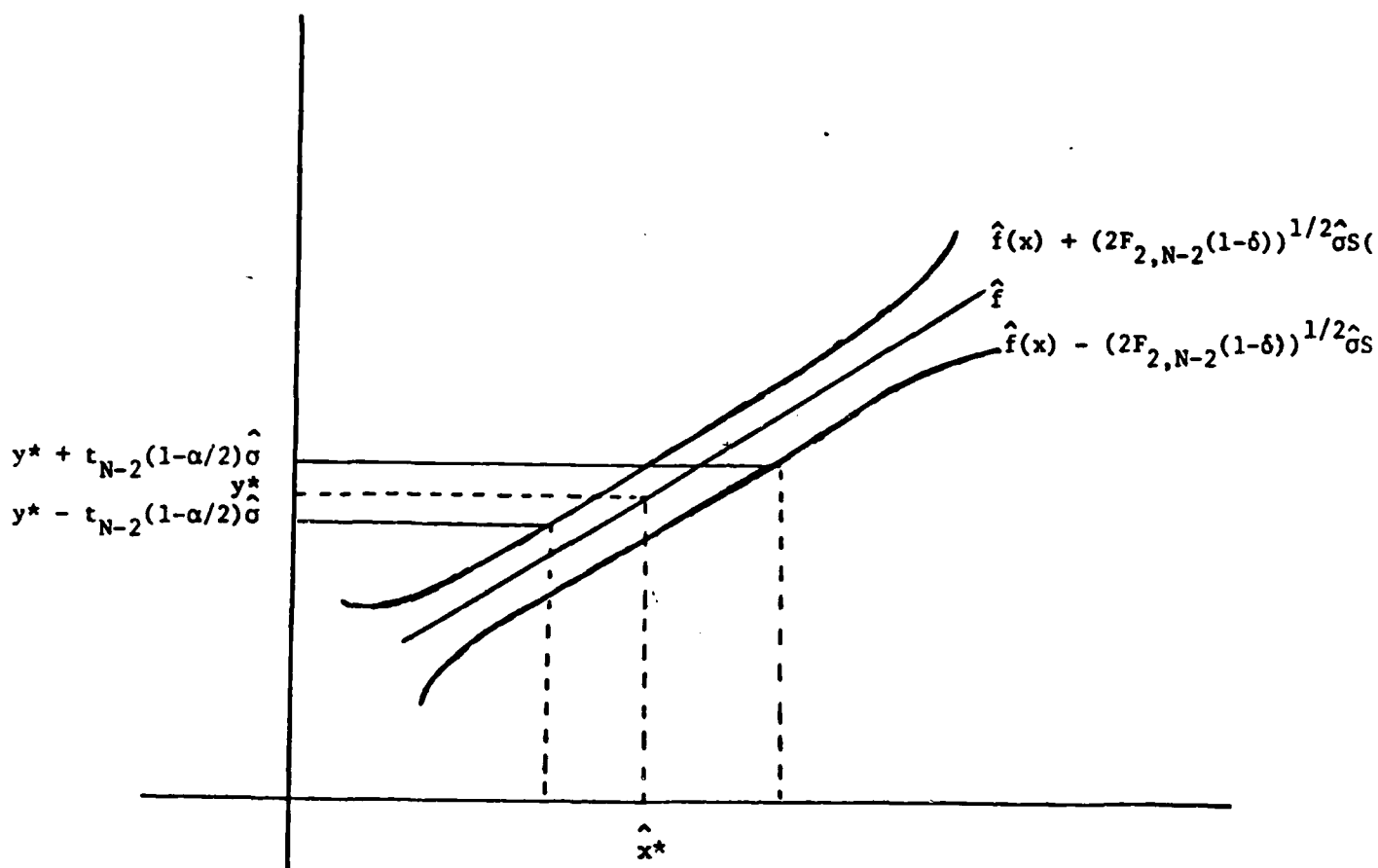
Calibration Intervals: Scheffe' Compared with Section 2 y = new observation x_L = lower endpoint of calibration interval x_U = upper endpoint Δ = length of calibration interval

First row in each entry is Scheffe' calibration; second row is calibration according to section 2; third row uses remark 2; fourth row uses remarks 1 and 2.

y	x_L	x_U	Δ	
.06	.0011	.0313	.0302	Scheffe'
	.0034	.0290	.0256	Section 2
	.0045	.0279	.0233	Remark 2
	.0051	.0273	.0222	Remarks 1&2
.10	.0592	.0883	.0292	
	.0615	.0860	.0245	
	.0626	.0849	.0223	
	.0631	.0845	.0214	
.15	.1314	.1601	.0287	
	.1337	.1578	.0240	
	.1348	.1566	.0218	
	.1353	.1562	.0209	
.22	.2316	.2614	.0298	
	.2340	.2591	.0251	
	.2351	.2579	.0229	
	.2356	.2574	.0219	
.30	.3453	.3780	.0327	
	.3476	.3757	.0281	
	.3487	.3745	.0258	
	.3494	.3738	.0244	
.38	.4586	.4949	.0363	
	.4608	.4926	.0318	
	.4619	.4914	.0295	
	.4629	.4904	.0275	

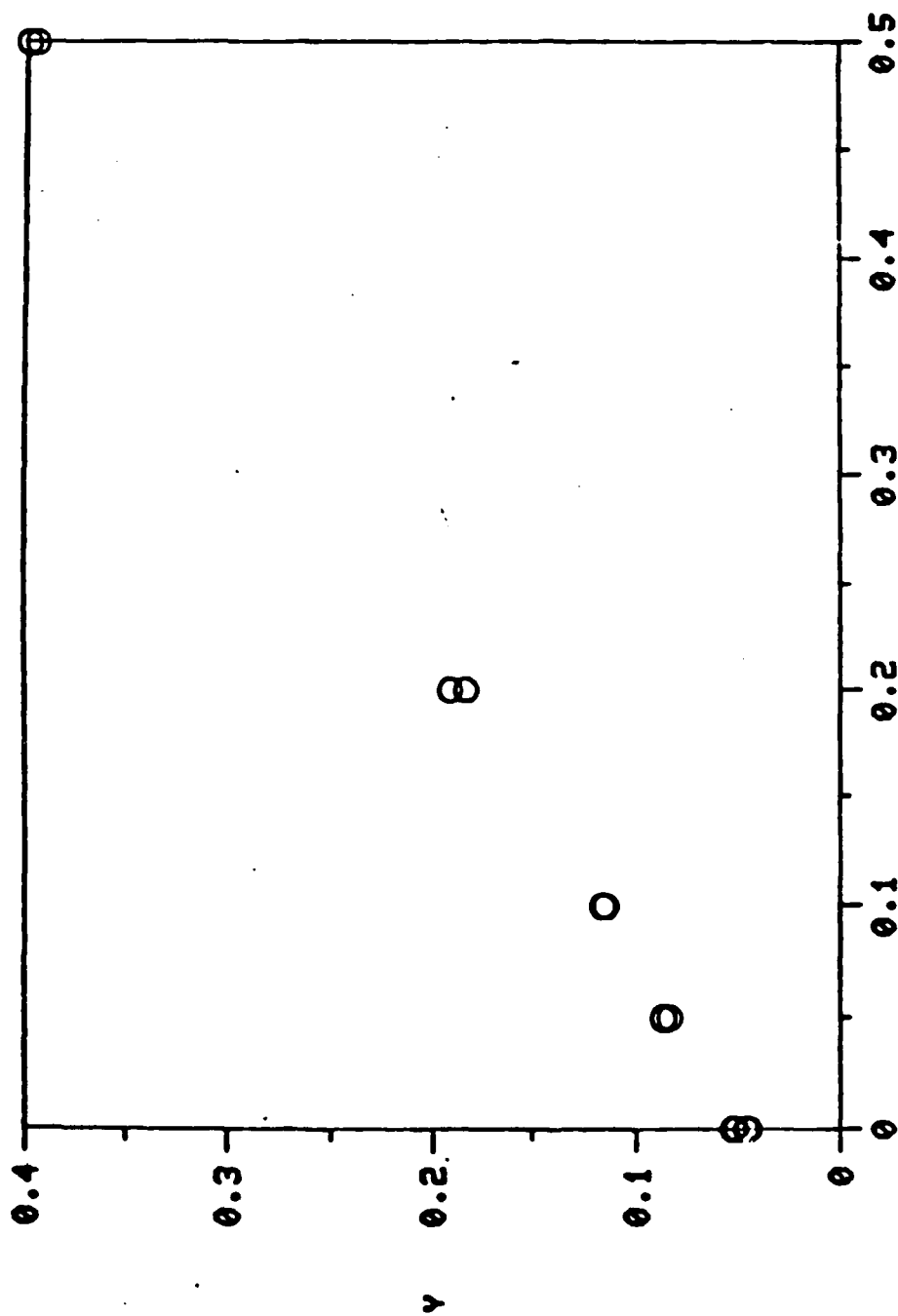
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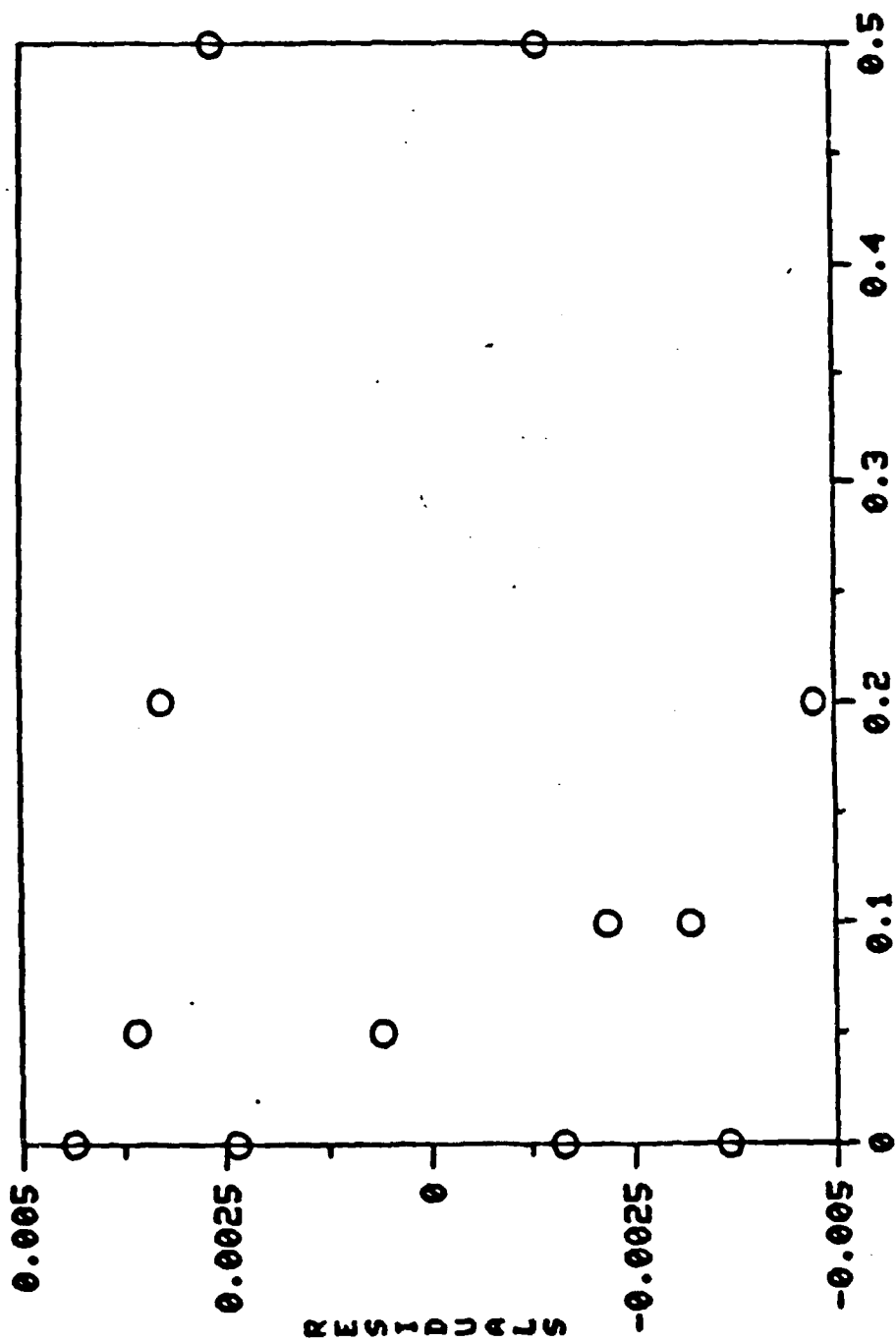


Interval for x^*

Figure 1



MICROGRAMS OF COPPER
IN DILUTE ACID SOLUTION: PLOT OF DATA
FIGURE 2a



MICROGRAMS OF COPPER
IN DILUTE ACID SOLUTION: RESIDUAL PLOT
FIGURE 24